भारतीय मानक
Indian Standard

IS 1448 (Part 115) : 2023

पेट्रोलियम और उसके उत्पाद — परीक्षण पद्धतियाँ

भाग 115 कच्चे तेल में नमक की मात्रा का निर्धारण

(पहला पुनरीक्षण)

Petroleum and Its Products — Methods of Test

Part 115 Determination of Salt Content in Crude Oil

(First Revision)

ICS 75.080

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October 2023 **Price Group 8**

Methods for Sampling and Test for Petroleum and Related Products of Natural or Synthetic Origin (Excluding Bitumen) Sectional Committee, PCD 01

FOREWORD

This Indian Standard (Part 115) (First Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the Methods for Sampling and Test for Petroleum and Related Products of Natural or Synthetic Origin (Excluding Bitumen) Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

This standard elucidates the procedure for determination of salt in crude oil. Salt present in crude oil recovered from reservoir have an adverse effect in downstream processes due to corrosion and catalyst deactivation. Pre-treatment operations are applied to the crude oil prior to transportation. Any crude oil to be shipped by pipeline, or by any other form of transportation, must meet strict regulations in regard to salt content. Thus, it is essential to measure the salt content in the crude oil for which this standard has been formulated.

Further, before the crude oil is introduced in the distillation column, it is necessary to remove the salt. Thus, knowing the measure of salt content.

The first revision has been brought out to keep pace with the latest technological developments and international practices. In this revision following major changes have been made:

a) Conductometric method, extraction method, and ion chromatography methods have been incorporated;
b) References have been updated.

The composition of the Committee, responsible for the formulation of this standard is given in Annex A.

In reporting the results of a test analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS 2 : 2022 ‘Rules for rounding off numerical values (*second revision*)’.

Indian Standard

PETROLEUM AND ITS PRODUCTS — METHODS OF TEST

PART 115 DETERMINATION OF SALT CONTENT IN CRUDE OIL

(First Revision)

1 SCOPE

1.1 This standard prescribes three test methods for determination of salt content in crude oil that is conductometric method, extraction method and ion chromatography method.

1.2 Method A covers the determination of the chloride (salts) concentration in crude oil. The range of concentration covered is 3.5 mg/kg to 500 mg/kg as chloride concentration/volume of crude oil.

1.2.1 This method measures conductivity in the crude due to presence of common chlorides, such as sodium, calcium, and magnesium. Other conductive materials present in the crude oil may also contribute to the conductivity.

1.3 Test method B is intended for the determination of total halide concentration of 0.002 percent to 0.02 percent, weight in crude petroleum, topped crude, residual cracking stock, and fuel oil. It may also be applied to the estimation of seawater contamination of used turbine oil and of marine diesel fuel.

1.4 Test method C for salt content in crude oil using extraction by hot water with mechanical stirring and detection by ION chromatography technique. The method covers the concentration of salt measurement start from 0.1 PTB and above.

2 REFERENCES

The standards given below contain provisions, which, through reference in text constitute provisions of this standard. At the time of publication, the editions indicated were valid. All the standards are subject to revision, and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent editions of these standards:

IS No.	*Title*
IS 1070 : 2023	Reagent grade water — Specification (*fourth revision*)
IS 1447 (Part 1) : 2021	Methods of sampling of petroleum and its products: Part 1 Manual sampling (*second revision*)
IS 1448 (Part 16) : 2014/ISO 3675 : 1998	Methods of test for petroleum and its products: Part 16 Crude petroleum and liquid petroleum products — Laboratory determination of density — Hydrometer method (*fourth revision*)
IS 4644 : 1968	Code of safety for benzene, toluene and xylene

3 METHOD A — CONDUCTIVITY METHOD

3.1 Principle

In conductometric test method, conductivity of a solution of crude oil in a mixed alcohol solvent is determined when an electrical stress is subjected. This test method measures conductivity due to the presence of inorganic chlorides, and other conductive material, in the crude oil. A homogenized test specimen is dissolved in a mixed alcohol solvent and placed in a test cell consisting of a beaker and a set of electrodes. A voltage is impressed on the electrodes, and the resulting current flow is determined using current measuring device. The chloride (salt) content is obtained by reference to a calibration curve of current versus chloride concentration of known mixtures or standard.

3.2 Apparatus and Glassware

3.2.1 The apparatus shall comprise of a conductivity unit capable of producing and displaying several voltage levels for applying stress to a set of electrodes suspended in a test beaker containing a test solution. The apparatus shall be capable of measuring and displaying the current (mA) conducted through the test solution between the electrodes at each voltage level.

> NOTE — Some apparatus is capable of measuring voltage and current internally and, after comparison to internal calibration curves, of displaying the resultant concentration.

3.2.2 *Pipette*, 10 ml — when the viscosity of crude oil is such that it is easy to transfer 10 ml sample completely, pipette may be used. In case viscosity of crude oil makes it difficult then a graduated cylinder may be used.

3.2.3 *Test Beakers*

3.2.4 *Cylinders* — 100 ml graduated, glass stoppered

3.2.5 *General Glassware*

3.3 Reagents

3.3.1 All reagents shall be free from halide.

3.3.2 *Wate*r — Grade 2 reagent water (*see* IS 1070)

3.3.3 *Mixed Alcohol Solvent* — mixture of 63 ml of 1-butanol and 37 ml of absolute methanol (anhydrous). To this mixture, add 3 ml of water.

CAUTION — The mixed alcohol solvent is flammable, can cause burning sensitization in the eyes. Its vapors are harmful, may be fatal or cause blindness.

> NOTE — The mixed alcohol solvent is suitable for use if its conductivity is less than 0.25 mA at 125 V AC (or 2 µS). High conductivity can be due to excess water in the solvent and it may indicate that the methyl alcohol used is not anhydrous.

3.3.4 *Hexanes, Reagent Grade*

CAUTION — Hexane is extremely flammable and harmful if inhaled.

> NOTE — Hexanes solvent is sometimes referred to or sold by other names, such as petroleum naphtha, petroleum ether, ligroine, petroleum benzine, and industrial naphtha.

3.3.5 *Calcium Chloride* (*$CaCl_2$*) *Solution* (*10 g/l*)

Transfer 1.00 g ± 0.01 g of $CaCl_2$, or the equivalent weight of a hydrated salt into a 100 ml volumetric flask and dissolve in 25 ml of water. Dilute to the mark with mixed alcohol solvent.

3.3.6 *Magnesium Chloride* (*$MgCl_2$*) *Solution* (*10 g/l*)

Transfer 1.00 g ± 0.01 g of $MgCl_2$, or the equivalent weight of a hydrated salt into 100 ml volumetric flask and dissolve in 25 ml of water. Dilute to the mark with mixed alcohol solvent.

3.3.7 *Sodium Chloride* (*NaCl) Solution* (*10 g/l*)

Transfer 1.00 g ± 0.01 g of NaCl into a 100 ml volumetric flask and dissolve in 25 ml of water. Dilute to the mark with mixed alcohol solvent.

3.3.8 *Oil, Refined Neutral*

Any refined chloride-free oil of approximately 20 mm^2/s (cSt) viscosity at 40 °C and free of additive.

3.3.9 *Mixed Salt Solution* (*Concentrated Solution*)

Combine 10.0 ml of the $CaCl_2$ solution, 20.0 ml of the $MgCl_2$ solution, and 70.0 ml of the NaCl solution, and mix thoroughly.

> NOTE — The proportion 10 : 20 : 70 is representative of the chlorides present in a number of common crude oils. When therelative proportions of calcium, magnesium, and sodium chlorides are known for a given crude oil, such proportions should be used for most the accurate results.

3.3.10 *Salts, Mixed Solution* (*Dilute Solution*) — Transfer 10 ml of the concentrated mixed chloride solution into a 1000 ml volumetric flask, and dilute to the mark with mixed alcohol solvent.

3.3.11 *Xylene* — reagent grade

CAUTION — Xylene is flammable and its vapors are harmful (*see* IS 4644).

3.4 Sampling

3.4.1 Obtain a sample in accordance with IS 1447 (Part 1). Sample homogeneity is to be ensured with a suitable mixer.

3.4.2 Samples with high viscous materials may be warmed until they are reasonably fluid before sampling; however, no sample shall be heated more than is necessary to lower the viscosity to a manageable level.

3.4.3 If samples of crude oil contain water and sediment and are in homogeneous by nature then presence of water and sediment will influence the conductivity of the sample. Utmost care shall be taken in obtaining homogenized representative samples.

3.5 Preparation of Apparatus

3.5.1 Support the apparatus on a level, steady surface, such as a table.

3.5.2 Prepare the apparatus for operation in accordance with the manufacturer's instructions for calibrating, checking, and operating the equipment.

CAUTION — The voltage applied to the electrodes can be as high as 250 V a.c., and hazardous.

3.5.3 Thoroughly clean and dry all parts of the apparatus, beakers, electrodes, and its accessories before starting the test, being sure to remove any solvent that had been used to clean the apparatus.

3.6 Calibration

3.6.1 Establish a blank measurement by following the procedure in **3.6.3** and **3.6.4,** omitting the mixed salts solution. When the indicated electrode current is greater than 0.25 mA at 125 V AC, water or another conductive impurity is present and its source must be found and eliminated before calibration can be completed. Determine a blank measurement each time with xylene or mixed solvent is used.

3.6.2 Take 15 ml xylene in a dry 100 ml measuring cylinder (**3.2.4**) using pipette (**3.2.2**). Add 10 ml of neutral oil and make up to 50 ml with xylene. Stopper and shake the cylinder vigorously for approximately 60 s to effect solution. Add a quantity of dilute mixed salts solution, in accordance with Table 1, that is appropriate to the range of salt contents to be measured. Dilute to 100 ml with mixed alcohol solvent. Again shake the cylinder vigorously for approximately 30 s to effect solution, and allow the solution to stand approximately 5 min. Pour the solution into a dry test beaker.

3.6.3 Immediately place the electrodes into the solution in the beaker, making sure that the upper edge of the electrode plates are below the surface of the solution. Adjust the indicated electrode voltage to a series of values, for example 25 V, 50 V, 125 V, 200 V, and 250 V AC. At each voltage, note the current reading and record the voltage displayed and the current to the nearest 0.01 mA. Remove the electrodes from the solution, rinse with xylene followed by hexane (*see* **3.3.4**), and allow them to dry.

Table 1 Standard Solution

(*Clause* 3.6.2)

Sl No. (1)	**Salt, g/m^3 of Crude Oil** (2)	**Salt, lb/1 000 bbl of Crude Oil** (3)	**Mixed Salts Solution (dilute),** ml (4)
i)	3	1.0	0.3
ii)	9	3.0	1.0
iii)	15	5.0	1.5
iv)	30	10.0	3.0
v)	45	16.0	4.5
vi)	60	21.0	6.0
vii)	75	26.0	8.0
viii)	90	31.0	9.5
ix)	115	40.0	12.0
x)	145	51.0	15.0
xi)	190	66.0	20.0
xii)	215	75.0	22.5
xiii)	245	86.00	25.5
xiv)	290	101.0	30.5
xv)	430	151.0	45.0

NOTE — With some apparatus, the detailed settings will not be required since the electronics are built-in for auto-ranging. Determination of the blank and the calibration standard responses are the same.

3.6.4 Repeat the procedure in **3.5.3**, using other volumes of mixed salts solution (dilute solution) as needed to cover the range of chloride contents of interest.

3.6.5 Subtract the value obtained for the blank measurement from the indicated current readings of each standard solution, and plot the chloride content against net current (mA) readings for each voltage on 3 by 3 cycle log-log paper, or other suitable format.

NOTES

1 The conductivity of solution is affected by the temperature of the specimen when measurements are made. The temperature of the test specimen at the time of measurement shall be within 3 °C of the temperature at which the calibration curves were made.

2 Some apparatus are capable of internally recording the current readings, standard concentration, and blank, and they provide an output in direct concentration units. Alternatively, some apparatus are also capable of internally converting and displaying the measured currents into conductivity units of micro Siemens, µS.

3 The apparatus are calibrated against standard solutions of neutral oil and mixed chloride solutions in xylene because of the extreme difficulties in keeping crude oil-brine mixtures homogeneous. The calibration may be confirmed, if desired, by careful replicate analysis of crude-oil samples by exhaustive extraction of salts with hot water, followed by titration of the chlorides in the extract.

4 In calibrating over a wide range of chloride concentrations, it may be necessary to apply several voltages to obtain current readings within the limit of the apparatus current level display (0 mA to 10 mA).

5 Higher voltages are applied for low concentrations and lower voltages are applied for high concentrations.

3.7 Procedure

Follow the procedure given in **3.6.3** taking crude oil in place of neutral oil and **3.6.4** to obtain voltage and current readings. Record the indicated electrode current to the nearest 0.01 mA and the nearest voltage. (*see* **3.6.5** Note 2).

3.8 Calculation and Report

3.8.1 Subtract the value obtained for the blank measurement from the value obtained from the specimen measurement to obtain the net current reading. From the calibration graph, read the indicated salt concentration corresponding to the net current (mA) reading of the sample (*see* **3.6.5,** Note 2).

3.8.2 Calculate the concentration in mg/kg by using the appropriate equation given below:

$$\text{Salt, mg/kg} = 1\,000 \times \frac{X}{d}$$

where

$$\text{Salt, mg/kg} = 2\,853 \times \frac{y}{d}$$

X = measured salt concentration, in g/m^3;
y = measured salt concentration, in PTB; and
d = specimen density, in kg/m^3, at 15 °C.

NOTE —The density of the specimen can be determined by IS 1448 (Part 16) or any other established density measurement methods.

3.8.3 Report the concentration directly in g/m^3 or lb/1 000 bbl.

3.9 Precision

3.9.1 Results of duplicate tests shall not differ by more than the following amounts:

Sl No. (1)	*Repeatability* (2)	*Reproducibility* (3)
i)	R (mg/kg) = 0.340 1 $X^{0.75}$	R (mg/kg) = 2.780 3 $X^{0.75}$
ii)	R (lb/1 000 bbl) = 0.253 1 $Y^{0.75}$	R (lb/1 000 bbl) = 2.069 $Y^{0.75}$

where

X = the average of two test results, mg/kg; and
Y = the average of two test results, lb/1 000 bbl (PTB).

4 METHOD B — EXTRACTION METHOD

4.1 Principle

In an extraction apparatus, the sample is extracted with water in the presence of a demulsifying agent and solvent. After removal of sulphide, the halides in the extract are determined volumetrically and the result is reported as sodium chloride.

4.2 Apparatus

4.2.1 *Extraction Apparatus* — given in Fig. 1

4.2.1.1 *Boiling flask* — 500 ml capacity

4.2.1.2 *Hopkins coil reflux condenser*

4.2.1.3 *Thistle funnel* — approximately 70 ml capacity, with indication for 50 ml level

4.2.1.4 *Heating tube*

4.2.1.5 *Heating coil*

4.2.1.6 *Filtering crucibles* — approximately 25 ml capacity: G-4 sintered glass or porcelain crucibles

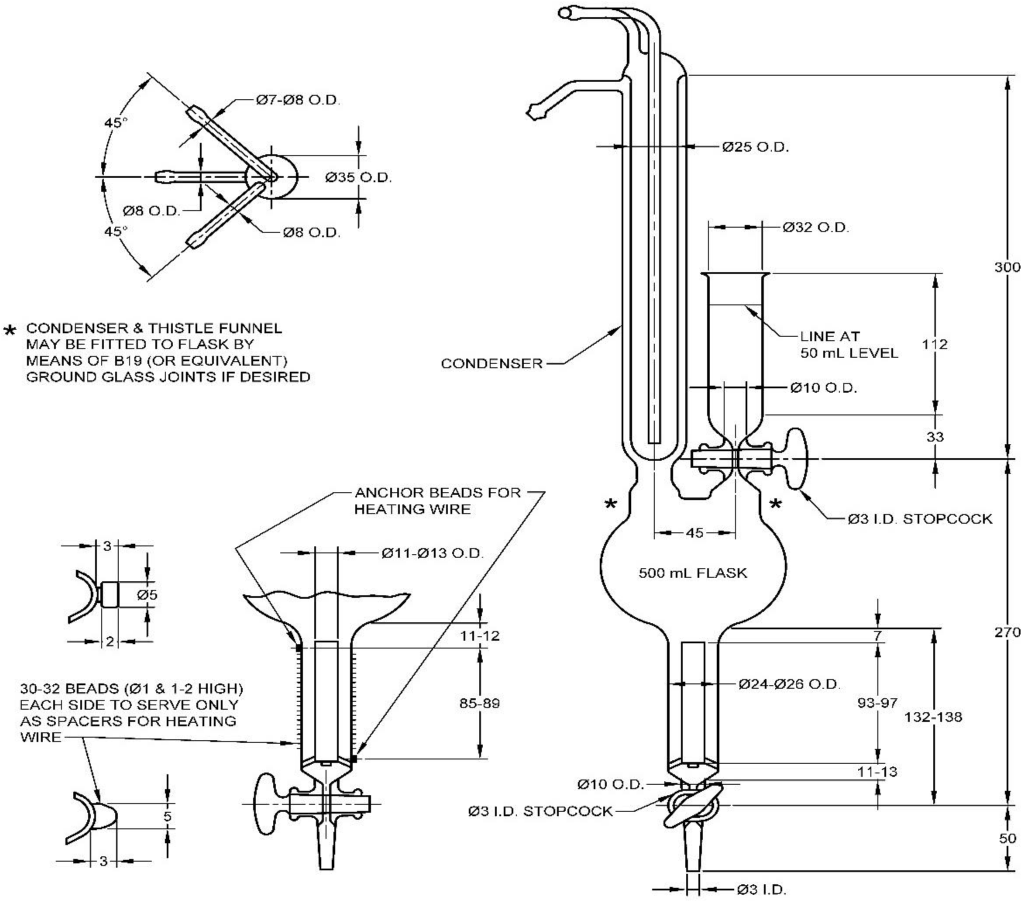


FIG. 1 EXTRACTION APPRATUS

4.2.1.7 *Erlenmeyer flask* — 500 ml, glass stoppered

4.3 Reagents

4.3.1 All reagents shall be free from halide.

4.3.2 *Acetone*

4.3.3 *Alcohol* — absolute ethanol, or denatured ethanol

4.3.4 3-*methylbutan-1-ol*

4.3.5 *Toluene*

4.3.6 *Lead Acetate Papers*

4.3.7 *Nitric Acid, 5 mol/l*

Dilute 1 volume of concentrated nitric acid with 2 volume of distilled water, and boil until it is colourless. Cool and blow the solution with air to remove oxides of nitrogen.

4.3.8 *Potassium Thiocyanate Standard Solution* — 0.05 mol/l

4.3.9 *Silver Nitrate Standard Solution* — 0.05 mol/l

4.3.10 *Ferric Alum Indicator*

Dissolve (350 ± 0.1) g of ferric ammonium sulphate in 1 litre of distilled water and add approximately 200 ml of 5 mol/l nitric acid.

4.4 Preparation of Sample

4.4.1 Follow the sampling procedure given in **3.4**.

4.5 Procedure

4.5.1 Weigh (80 ± 0.5) g of the sample into a 250 ml beaker and heat it to 60 °C ± 0.5 °C. Heat approximately 40 ml of toluene to the same temperature and add it slowly to the sample with constant stirring until solution is completely homogenized. Transfer this solution quantitatively to the flask of the extraction apparatus through the thistle funnel, and wash it with two part of 15 ml of toluene, each of about 60 °C. Without allowing the mixture in the flask to cool, add 25 ml of hot alcohol and 15 ml of hot acetone.

4.5.2 Boil the mixture vigorously for 2 min, add (125 ± 1) ml of distilled water after the boiling has come to end. Again boil the mixture for a further 15 min.

4.5.3 Allow the phases to cool and separate. Draw off the lower layer and filter it using a Whatman No. 41 or equivalent filter paper, if necessary. The volume of extract available is taken as 160 ml because some alcohol and acetone remain dissolved in the toluene phase. Take (100 ± 1) ml of the extract in a beaker and add approximately 5 ml of the nitric acid. Cover the beaker with a watch glass and boil the content. Periodically test the vapours for Hydrogen sulphide with lead-acetate paper and continue to boil for a further 5 min after a negative results is obtained. Cool and rinse the contents of the beaker into a titration flask with distilled water. Add approximately 10 ml of 3-methylbutan-1-ol and approximately 3 ml of ferric alum indicator

4.5.4 Add 0.5 ml of the thiocyanate solution. Titrate the mixture with silver nitrate solution to a colourless end point, agitating the contents of the flask continuously, and add an excess of 5 ml of the silver nitrate. Stopper the flask and shake it vigorously for 15 s to coagulate the precipitate.

Titrate slowly with the thiocyanate solution until the end point is reached when indicated by slower fading of the red coloration. Shake the flask again and continue the titration dropwise until a red coloration no longer disappears on vigorous shaking.

4.5.5 Make a blank titration by repeating the procedure, replacing the 80 g of sample with 95 ml of toluene.

4.6 Calculation and Report

4.6.1 Calculate the halide content of the sample either as percentage sodium chloride by weight, or mg per litre.

NaCl, percent by weight =

$$\frac{9.35\ [(V_1 - V_2)\ M - (V_3 - V_4)\ m]}{W}$$

NaCl, mg per litre =

$$\frac{9.35 \times 104\ G\ [(V_1 - V_2)\ M - (V_3 - V_4)\ m]}{W}$$

where

V_1 = volume of silver nitrate solution consumed to titrate the sample;

V_2 = volume of silver nitrate solution consumed to titrate the blank;

M = molarity of the silver nitrate solution;

V_3 = volume of thiocyanate solution consumed to titrate the sample, including the 0.5 ml added **4.5.4**;

V_4 = volume of thiocyanate solution consumed to titrate the blank, including the 0.5 ml added **4.5.4**;

m = molarity of the thiocyanate solution;

W = mass, in g, of sample; and

G = density of the sample at 15 °C.

4.6.2 Report the result to the nearest 0.002 percent or to the nearest 2 mg per litre as the salt content.

4.7 Precision

4.7.2 Results of duplicate test should not differ by more than:

Sl No. (1)	*Repeatability* (2)	*Reproducibility* (3)
i)	0.002 percent	0.004 percent
ii)	12 mg/l	24 mg/l

5 METHOD C (ION CHROMATOGRAPHY METHOD)

5.1 Principle

An extracted salt by hot water extraction from crude sample is injected into a stream of sodium hydroxide eluent and passed through an ion exchanger column. The anions are separated on the basis of their relative affinities for low capacity, strongly basic anion exchangers. The separated anions are directed onto an anion self-regenerating suppresser (ASRS) having cation exchange membrane. In the suppresser the separated anions are converted to their highly conductive acid forms. The separated anions in their acid forms are measured by conductivity detector and identified on the basis of retention time as compared to standard.

5.2 Reagents and Materials

5.2.1 *Nitrogen, Purity* — minimum 99.95 mol percent
.

5.2.2 *Sodium Hydroxide* — purity, minimum 98 percent

5.2.3 *Toluene, Purity* — minimum 99 percent

5.2.4 *Isopropyl Alcohol*— purity, minimum 99 percent

5.2.5 *Deionized Water or Grade* 1 *Water of IS* 1070

5.2.6 *Standard for Calibration and Identification* — Sodium chloride ultrapure, purity, minimum 99 percent.

5.2.7 *Volumetric Glassware* — Class A pipettes, burettes, one-mark volumetric flasks and measuring cylinders as required for the preparation of solutions and standard blends.

5.2.8 *Sample Vials* — 2 ml glass/plastic vials with filter cap for auto sampler

5.2.9 *Syringe* — Plastic syringe, 5 ml capacity

5.2.10 *Filter Paper* — Whatman No. 42 filter paper and 0.2 µm syringe filter or equivalent qualitative filter paper.

NOTE — All chemicals shall be chloride free except sodium chloride.

5.3 Apparatus

5.3.1 *Ion Chromatographic System*

5.3.1.1 Ion chromatographic system shall consist of the following components:

a) Eluent reservoirs, and a degassing unit for two eluents;

b) Pump, suitable for step gradient technique;

c) Sample delivery device (for example, sample pump) including a sample injection system incorporating a sample loop of appropriate volume or auto sampler device;

d) Column switching valves (for example, 6 port valve) including a device for timing and controlling valves and pump;

e) Concentrator column (may be required for low concentrations);

f) Separator column;

g) Conductivity detector with an anion suppressor device assembly; and

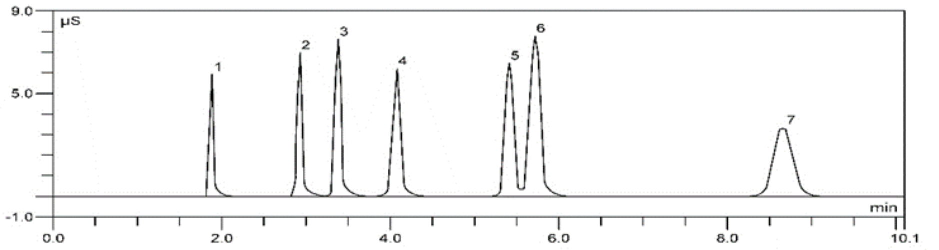
h) Recording device (for example, recorder, integrator with printer, PC with software for data acquisition and evaluation).

5.3.1.2 Ion chromatographic system shall be capable of resolving the individual salts (*see* Table 2 and Fig. 2) when operated at the conditions given in Table 3

Table 2 Retention Time and Sequence of Anions for Reference

(*Clause* 5.3.1.2)

Sl No. (1)	Component (2)	Retention Time, in min (Approx.) (3)
i)	Fluoride	1.87
ii)	Chloride	2.91
iii)	Nitrite	3.37
iv)	Sulphate	4.06
v)	Bromide	5.39
vi)	Nitrate	5.69
vii)	Posphate	8.66



Sl No. (1)	**Peak Number** (2)	**Component** (3)
i)	1	Fluoride
ii)	2	Chloride
iii)	3	Nitrite
iv)	4	Sulphate
v)	5	Bromide
vi)	6	Nitrate
vii)	7	Phosphate

FIG. 2 REFERENCE CHROMATOGRPH OF CALIBRATION STANDARDS FOR THE RESOLUTION OF ANIONS

Table 3 Operating Conditions for Reference

(*Clause* 5.3.1.2)

Sl No. (1)	**Component** (2)	**Conditions** (3)
i)	Analytical column	AS11-HC (4 × 200 mm) or equivalent
ii)	Guard column	AG-HC (4 × 50 mm) or equivalent.
iii)	Eluent	30 mM NaOH
iv)	Temperature	30 °C
v)	Eluent flow rate	1.5 ml/min
vi)	Suppressor	Anion self-regenerating suppresser
vii)	Detector	Conductivity cell
viii)	Injection loop	10 µl (5 µl to 200 µl capacity)
ix)	Calibration	External standard traceable to NIST

5.3.2 Overhead mechanical stirrer (as shown in Fig. 3).

5.3.3 Glass container designed for hot water circulation (as shown in Fig. 3).

5.3.4 *Hot Water Circulating Bath* — capable to maintaining temperature at 60 °C ± 2 °C

5.3.5 *Analytical Balance* — capable of weighing to the nearest 0.1 mg

5.3.6 *Filtration Assembly* — capable to filter solution through 0.8 micron membrane filter under vacuum.

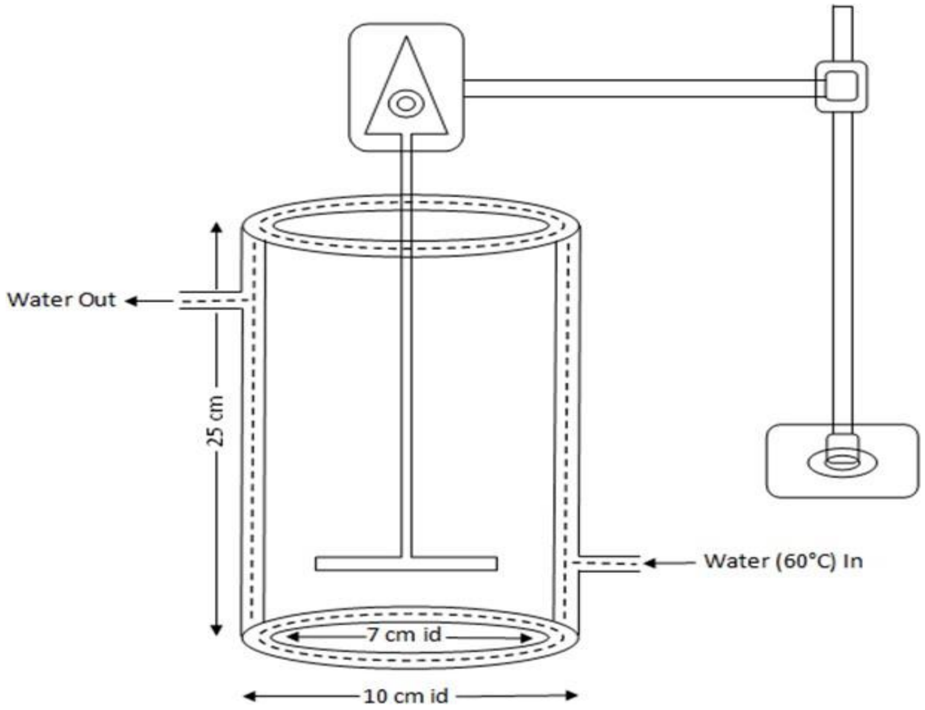


FIG. 3 HOT WATER CIRCULATION GLASS CONTAINER WITH OVERHEAD MECHANICAL STIRRER

5.4 Sampling

Follow the sampling procedure given in **3.4**

5.5 Preparation of Calibration Standards

Use certified reference material or prepare chloride standard in laboratory by following procedure.

5.5.1 *Preparation of Chloride Stock Solution* 1 000 *mg/l*

Dissolve 1.6485 gm ± 0.000 1 gm of previously dried at 105 °C ± 1 °C sodium chloride in 250 ml of deionized water or grade 1 water of IS 1070 in 1 000 ml volumetric flask and dilute up to the mark with deionized water or Grade 1 water of IS 1070.

5.5.2 *Preparation of Standard Solution of Chloride* 100 *mg/*l

Pipette out 100 ml of 1 000 mg/l chloride stock solution into 1 000 ml volumetric flask. Make up to the mark with deionized water or grade 1 water of IS 1070.

5.5.3 *Preparation of Standard Solution of Chloride* 10 *mg/l*

Pipette out 100 ml of 100 mg/l chloride standard solution into 1 000 ml volumetric flask. Make up to the mark with deionized water or grade 1 water of IS 1070.

5.5.4 Prepare 0.1 mg/l, 1 mg/l and 5 mg/l chloride working standard solution from 10 mg/l chloride by further dilution with deionized water or Grade 1 water of IS 1070.

NOTE — Add more level of working standards for calibration plot as per salt content estimation in sample.

5.5 Eluent and Regenerant

5.5.1 *Eluent, 30 mM NaOH*

Weigh 2.4 g of 50 percent (w/w) sodium hydroxide and make up to 1000 ml in volumetric flask with degassed, deionized water or Grade 1 water of IS 1070.

5.5.2 *Regenerant,* 1 *M HCl*

5.6 Calibration

5.6.1 Set up the ion chromatographic system according to the instrument manufacturer's instructions and operational conditions described in Table 3. Run the starting eluent, once the baseline is stable analysis can begin. If metal clean up, concentrator columns and suppressor devices are being used, regenerate according to the instrument manufacturer's instructions before use.

NOTE — If different columns with different diameter are used then adjust the optimum flow and configure chromatographic system as per instrument manufacturer's instructions and as per requirement. Set required flow of nitrogen gas (for water degassing) by using flow meter.

5.6.2 Inject the prepared calibration standards as per the sample measurement requirement. Use required range of standards for level calibration. Determine and record the retention times for all component to be evaluated. Calculate the calibration factor for all the components to be evaluated, using below equation:

$$f_i = \frac{m_i}{A_i}$$

where

f_i = calibration factor;
i = components;
m_i = mass of component in calibration standard; and
A_i = peak area of component in calibration standard.

5.7 Quality Control

Run a blank and any mid-range standard on the prepared calibration baseline. The observed results shall remain within acceptable precision limit of this method. In case deviation from the precision limit is observed, recalibrate the instrument.

5.8 Procedure

5.8.1 *Sample Preparation*

Procedure for preparation of sample has been divided into two parts as per the concentration of the salt.

5.8.1.1 *Low salt crude sample preparation* (*for the measurement range of* 0.1 *PTB* to 3 *PTB*)

5.8.1.1.1 Switch ON the hot water circulating bath and set the temperature at 60 °C.

5.8.1.1.2 Mix the sample vigorously using overhead mechanical stirrer for 5 min and take 100 ml of homogenized sample in specially designed glass container having hot water circulation.

5.8.1.1.3 Add 100 ml toluene into it and mix for 1 min, add 100 ml of hot deionized water or Grade 1 water of IS 1070. Mix the mixture by using mechanical stirrer with minimal rpm to ensure sufficient mixing for 30 min. Ensure no spillover takes place in glass container during stirring. Allow to separate hydrocarbon and water layers.

> NOTE — In case aqueous layer and crude oil layer are not getting separated then transfer around 100 ml of bottom emulsion to a hard glass tube and keep the tube in oil bath at 100 °C for 30 min. Collect the aqueous layer from bottom of the tube using a pipette.

5.8.1.1.4 Collect all aqueous layer from bottom in beaker by filtering through 2.5 µm ash less filter paper or equivalent. Refilter it through 0.2 µm syringe filter before injecting into ion chromatographic instrument.

5.8.1.2 *High salt crude sample preparation* (*for the measurement range above* 3 *PTB*)

5.8.1.2.1 Mix the sample vigorously in original sample container by overhead mechanical stirrer for 5 min. Take 10 ml or appropriate aliquot as per salt estimation of homogenized sample in specially designed glass container having hot water circulation and follow the steps from **5.8.1.1.3** to **5.8.1.1.4.**

> NOTE — Use dilution factor 10 for 10 ml sample or utilize as per selected volume of sample.

5.8.1.3 Prepare a blank solution without using sample by following steps from **5.8.1.1.3** to **5.8.1.1.4**.

5.8.2 *Chromatographic Analysis*

5.8.2.1 Inject prepared aqueous aliquot of blank and sample into the ion chromatograph. Obtain a chromatogram with integrated peak report presenting the retention times and integrated area of detected component.

5.8.2.2 Compare the retention times and area of sample components with calibration analysis for identification and quantification of chloride present.

5.8.3 *Calculation*

5.8.3.1 *Chloride* (*ppm*)

Instrument software calculate automatically or calculate manually using following equation;

$$\text{Chloride (ppm)}, S = f_{Cl} \times A_S \times d$$

where

f_{Cl} = calibration factor of chloride ;
A_S = peak area of; chloride in sample; and
d = dilution factor.

> NOTE — Dilution factor need to be applied only in case of sample quantity is taken less than 100 ml for high salt. for example, if sample quantity is taken 10 ml than dilution factor is 10.

5.8.3.2 *Chloride* (*PTB*)

$$\text{Salt Crude (PTB)} = (S - B) \times 0.578$$

where

S = chloride present in sample, in ppm;
B = chloride present in blank, in ppm;
0.578 = conversion factor to convert chloride ppm to salt as NaCl

PTB; and
PTB = pound per thousand barrel.

NOTE — Factor 0.578 is derived from below calculation.

$$0.578 = \frac{\text{Molecular weight of Sodium Chloride}}{\text{Molecular weight of Chlorine}} \times 0.350\,9$$

where

1 ppm is equivalent to 0.350 9 PTB to convert salt result from ppm to PTB.

5.9 Reporting

5.9.1 Report the salt content to the nearest 0.1 PTB

5.9.2 For concentration < 0.1 of salt, report as '< 0.1 NaCl PTB'.

5.10 Precision

Precision at 95 percent confident level is given in Table 4.

5.10.1 *Repeatability* (*r*)

The difference between two successive test results obtained by the same operator with the same apparatus under constant operating conditions on identical test material would be in the normal and correct operation of the test method.

5.11 Intermediate Precision (R)

The difference between two test results independently obtained by different operators working in samelaboratories on nominally identical test material would be in the normal and correct operation of the test method.

5.12 Reproducibility — It is yet to be determine.

Table 4 Precision at 95 Percent Confident Level

(*Clause* 5.10)

Sl No. (1)	Salt in PTB (2)	Repeatability (r) (3)	Intermediate Precision (R) (4)
i)	0.59	0.032	0.064
ii)	16.89	0.198	2.238

ANNEX A

(*Foreword*)

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Methods for Sampling and Test for Petroleum and Related Products
of Natural or Synthetic Origin (Excluding Bitumen) Sectional Committee, PCD 01

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